LABORATORY DATA AND DATA PACKAGE REVIEW

(Technical and Administrative Review)

Project number: VP0973	Analyst: J. Suggs	Reviewer: B. Burns
Project name: Western Zirconium		
Technique: XRF	PAC: R. Ross	Date submitted for review: 02/07/12 original, 04/10/12

Administrative and Technical Review

Data package includes:		Verification:	
X	Objective of work	X	Proofread for content
X	List of samples analyzed	X	Check spelling and punctuation in text
X	Method reference(s)	X	Check for data transcription errors
X	Description of sample prep/subsampling	X	Check spelling of compounds/analytes
X	Instrument identification	X	Project number, analyst's initials and date on each NEIC generated record
X	Software (include version number)	X	Instrument Logbook entry by analyst
X	QC requirements/data quality summary	N/A	Reference to and description of tables
X	Date of analysis		
X	Summary of results		

Administrative and technical findings summary and actions requested, if any. Minor corrections, for example things noted by sticky note, need not be listed:*

PAC found error in data summary: "chloride" used in sentence instead of "chlorine"

PAC requested correction to the data summary.

Reviewer's signature: [signature] **Date:** 4/10/12

Response to Action(s) Requested*

Analyst's signature: J Suggs **Date:** 4/10/12

Final Statement of Reviewer

I have reviewed this data package and it meets the documentation requirements of the NEIC Quality System. The data support the results being reported.

Reviewer's signature: [signature] **Date:** 4/10/12

I have presented a summary of QC data from these analyses to a Branch QA representative. N/A

Data were provided by: email _____ hardcopy _____ LIMS _____

Analyst's signature: J Suggs **Date:** 4/10/12

*Attach additional sheets as necessary.

LABORATORY DATA AND DATA PACKAGE REVIEW

(Technical and Administrative Review)

Project number: VP0973	Analyst: J. Suggs	Reviewer: B. Burns
Project name: Western Zirconium		
Technique: XRF Qualitative	PAC: R. Ross	Date submitted for review: 02/07/12

Administrative and Technical Review

Data package includes:		Verification:	
X	Objective of work	X	Proofread for content
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X	Date of analysis		
X	Summary of results		

Administrative and technical findings summary and actions requested, if any. Minor corrections, for example things noted by sticky note, need not be listed:*

SIMPLE EDITORIAL SUGGESTIONS. MINOR CODE MISSING ON CERTAIN ITEMS

Reviewer's signature: [signature] **Date:** 2/13/12

Response to Action(s) Requested*

Corrections made to indicated items.

Analyst's signature: Jennifer Suggs **Date:** 2/13/12

Final Statement of Reviewer

I have reviewed this data package and it meets the documentation requirements of the NEIC Quality System. The data support the results being reported.

Reviewer's signature: [signature] **Date:** 2/14/12

I have presented a summary of QC data from these analyses to a Branch QA representative. N/A

Data were provided by: email _____ hardcopy _____ LIMS _____

Analyst's signature: Jennifer Suggs **Date:** 2/14/12

*Attach additional sheets as necessary.

VP0973 – Western Zirconium

April 10, 2012
J. Suggs

X-ray Fluorescence Results
Qualitative Analysis

Five samples, labeled 30177-5, 30178-5, 30179-1, 30179-5, and 30180-3, were received for XRF qualitative analysis. Elements of interest in the analysis were sulfur, zirconium, hafnium, and the Resource Conservation and Recovery Act metals (arsenic, barium, cadmium, chromium, lead, selenium, and silver). Determinations were made on the Rigaku ZSX 101e spectrometer operated by the ZSX program (version 3.36). The NEIC operating procedure followed was NEICPROC/02-003R2, X-ray Fluorescence Spectrometry (XRF) Using the Rigaku ZSX 101e. Analysis was performed in January, 2012.

Sample Preparation

The samples were flaky solids that ranged in color from a clear white to gray. Use of a mortar and pestle to crush some of the sample material was ineffective. Therefore, all samples, a magnesium chloride standard, and a boric acid blank were prepared using the Spex Freezer Mill. Approximately 3 to 4 g of material was placed in a freezer mill tube and was agitated in the liquid nitrogen bath for 4 to 5 minutes. This process was repeated until approximately 25 g of the crushed material was collected.

Samples were pressed into 40 mm pellets with boric acid as the side and backing material. Approximately 5 g of sample was used for each pressed sample pellet. A boric acid pellet was prepared as a blank and a magnesium chloride standard (Fisher Scientific, lot #021053) and a NIST 2711 standard reference material (SRM) pellet were prepared for quality control .

Analysis

The standard, blank, and samples were analyzed using the Rigaku fundamental parameters program EZscan. The program scans for elements from fluorine through uranium in the periodic table and identifies the peaks. The short scan was selected due to concerns about the stability of the sample pellets.

Raw data was processed using the SQX data processing component of the ZSX program. Results for the magnesium chloride standard and the samples were processed using water as the balance formula. The NIST 2711 SRM data were processed using oxygen as the balance element. Raw data of the boric acid blank was processed using the formula of boric acid as the balance element (H_3BO_3).

Results

Concentrations for magnesium and chlorine for the samples were very similar to the magnesium chloride standard. The analysis showed the presence of zirconium as a minor element (0.1% to 5%) in sample 30178-5, and as a trace element (< 0.1%) in samples 30180-3, 30179-1, 30177-5, and 30179-5. Barium was identified by the instrument as a trace element in the 30178-5 analytical original and duplicate, but the peak was also visible in the spectrum of the analytical triplicate. Barium was also identified as a trace element in 30179-1 and 30179-5.

Quality Control

Quality control included a boric acid blank pellet, a magnesium chloride pellet, an analytical triplicate pressed from sample 30178-5, and a standard reference material containing elements of interest in minor and trace amounts. NIST 2711 is certified for barium and lead as a minor elements and sulfur, arsenic, cadmium, selenium and silver as trace elements; chromium, hafnium, and zirconium are listed as non-certified trace elements. Results from the NIST 2711 SRM showed reproducibility of

measurement. Results from the analytical triplicate showed agreement and indicated that the freezer mill sample preparation yielded a uniform sample for subsampling.

Uncertainty

The analytical results are qualitative only. Sulfur, lead, barium, zirconium, and arsenic were correctly identified in the NIST 2711 SRM. Barium Kα and zirconium Kα lines were identified in the spectra for samples containing those elements.

Name	Project No. Location	56232
J. Suggs	VP0973	Date: 1-5-12

pellet pressing bal #4430

#30177-5 after freezer mill prep on 1-4-12 5.001g

10.04 g boric acid backing

1-18

#30179-1	5.008 g	10.13 g boric acid backing
30180-3	5.002 g	10.02g boric acid
30178-5	5.009 g	10.08 g boric acid
30178-5 dup	5.003 g	10.06 g boric acid
30178-5 trip	5.93 g	10.04 g boric acid

1-23-12

boric acid freezer mill prep	5.007 g	10.02g boric acid backing
#30179-5	5.007g	10.02g " " "
#30177-5	5.005g	10.05g " " "
$MgCl_2$ freezer mill prep	5.007g	10.02g " " "

MgCl - Fisher Scientific Lot #021053

$\cdot 6H_2O$

JAS

Name	Project No. Location	56234
J. Suggs	JP0973 for multiple projects	Date: 1-19-12

XRF Pellets – Standards & Blank for Repeated Use

Boric Acid Blank 15.006g
EMD Boric Acid Crystals Lot # 47288752

NIST 2710 Montana Soil SRM 5.004g + 10.02 boric acid backing

1-23-12

NIST 2711 Montana Soil SRM 5.004g + 10.02 boric acid

JMS

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2711

Montana Soil

Moderately Elevated Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. SRM 2711 is a moderately contaminated soil that was oven-dried, sieved, radiation sterilized, and blended to achieve a high degree of homogeneity. A unit of SRM 2711 consists of 50 g of the dried material.

The certified elements for SRM 2711 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should **NOT** be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

NOTICE AND WARNINGS TO USERS

Expiration of Certification: This certification of SRM 2711 is valid, within the measurement uncertainties specified, until **31 December 2011**, provided the SRM is handled in accordance with instructions given in this certificate (see *Instructions for Use*). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The overall direction and coordination of the analyses were under the chairmanship of M.S. Epstein and R.L. Watters, Jr. of the NIST Inorganic Analytical Research Division.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by T.E. Gills and J.S. Kane. Revision of this certificate was coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 18 July 2003
See Certificate Revision History on Page 6

John Rumble, Jr., Chief
Measurement Services Division

VP0973 JAS 1-31-12

Table 1. Certified Values

Element	Mass Fraction (%)		Element	Mass Fraction (µg/g)	
Aluminum	6.53	± 0.09	Antimony	19.4	± 1.8
Calcium	2.88	± 0.08	Arsenic	105	± 8
Iron	2.89	± 0.06	Barium	726	± 38
Magnesium	1.05	± 0.03	Cadmium	41.70	± 0.25
Phosphorus	0.086	± 0.007	Copper	114	± 2
Potassium	2.45	± 0.08	Lead	1162	± 31
Silicon	30.44	± 0.19	Manganese	638	± 28
Sodium	1.14	± 0.03	Mercury	6.25	± 0.19
Sulfur	0.042	± 0.001	Nickel	20.6	± 1.1
Titanium	0.306	± 0.023	Selenium	1.52	± 0.14
			Silver	4.63	± 0.39
			Strontium	245.3	± 0.7
			Thallium	2.47	± 0.15
			Vanadium	81.6	± 2.9
			Zinc	350.4	± 4.8

Noncertified Values: Noncertified values, shown below, are provided for information only. An element concentration value may not be certified, if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Carbon	2	Bromine	5
		Cerium	69
		Cesium	6.1
		Chromium	47
		Cobalt	10
		Dysprosium	5.6
		Europium	1.1
		Gallium	15
		Gold	.03
		Hafnium	7.3
		Holmium	1
		Indium	1.1
		Iodine	3
		Lanthanum	40
		Molybdenum	1.6
		Neodymium	31
		Rubidium	110
		Samarium	5.9
		Scandium	9
		Thorium	14
		Tungsten	3
		Uranium	2.6
		Ytterbium	2.7
		Yttrium	25
		Zirconium	230